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STUDY OF PHARMACEUTICAL MANUFACTURING WASTEWATER TREATMENT SYSTEM AND AERATED TREATMENT SYSTEM

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Nebraska University
Lincoln, Nebraska

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By

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STUDY OF PHARMACEUTICAL MANUFACTURING WASTEWATER CHARACTERISTICS AND AERATED TREATMENT SYSTEM

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INTRODUCTION

The characteristics and treatability of wastewaters from pharmaceutical manufacturing and processing plants vary considerably depending upon the type of products being produced. Treatment of these wastewaters is complicated by the variability in the wastewater characteristics and by the nature of many of the products produced. The material presented in this paper was obtained from a study which was made of the wastewaters and the aerated treatment system for Dorsey Laboratories, a pharmaceutical formulation plant located at Lincoln, Nebraska.

The Dorsey Laboratories plant is located a considerable distance from a municipal sanitary sewer system. As a result, all wastewaters from the plant, except for cooling and boiler water, are treated in an extended aeration system prior to discharge to a receiving stream. The wastewaters include sanitary wastes, wastes from a company operated cafeteria, and wastes from production and cleanup operations. Wastewater from cooling and boiler operation is discharged to a holding pond

which also serves as an emergency source of water for fire fighting purposes.

The pharmaceutical products produced at Dorsey Laboratories are primarily the non-prescription type and may be in either the liquid or tablet form. Their uses include sedatives, digestive aids, and medication for arthritis, coughs, colds, hay fever, and sinus and bacterial infections. Production is on a batch basis and the type product produced varies from day to day. The plant employs approximately 225 people and generally operates on a single shift, 40 hour, 5-day week.

The treatment system consists of a factory-built extended aeration plant. It is not provided with facilities for wasting excess sludge nor for the positive return of sludge to the aeration basin, however, slots or channels are provided for sludge return. The capacities of the aeration basin and the settling basin are 21,000 gallons and 1935 gallons, respectively. The design detention period for the aeration basin was 25 hours. The design organic loading was 68 lb of BOD per day. The plant is equipped with two air compressors, each capable of delivering 120 cfm of standard air at 3.5 psi pressure.

SCOPE OF THE STUDY

The wastewater treatment plant has been studied periodically since June 1965. These studies indicated that the plant was not producing a satisfactory effluent. In addition, operational problems such as bulking sludge, excessive loss of solids from the system, and a concentrating of solids in the clarification basin were often

encountered. The study which is reported in this paper was conducted during 1968 and 1969.

The study was conducted in three phases. These were: (1) an initial evaluation of the existing treatment system and the wastewater characteristics; (2) an evaluation of the treatability of the wastewater in an extended aeration system in the laboratory under closely controlled conditions; and (3) the incorporation of the findings in the laboratory in a more detailed field evaluation of the existing treatment system.

INITIAL FIELD STUDY

During the first phase of this study three, one-day composites were made of the influent and the effluent from the wastewater treatment facilities. Some of the data obtained from these composites are presented in Table I. In interpreting this data, it should be remembered that the plant normally operates on a single 8-hour shift. Thus, the indicated daily hydraulic and organic loadings are actually received at the treatment plant during an 8-10 hour period. There are no provisions for equalizing the flow rate to the treatment plant.

The variability in the loading on the treatment plant is apparent from the high maximum flow rates obtained during these composite periods and the corresponding long detention times in the aeration basin. Although there was generally some reduction in the total solids concentration in the wastewater passing through the plant, the concentrations of total and volatile suspended solids in the effluent was much higher than that in the raw wastewater. The percentage reduction in BOD obtained in the treatment plant ranged from 30 to 70 for the composite

Table I

Composite Data - Initial Field Study

	Date of Composite - 1968		
	Wednesday January 3	Tuesday February 13	Tuesday March 19
Flow, gal/day	6,200	5,400	15,400
max. rate, gal/day	29,000	28,300	45,600
Detention time, days	3.4	3.9	1.4
Total Solids, mg/l			
Influent	1,270	2,130	868
Effluent	1,410	1,300	725
Total Volatile Solids, mg/l			
Influent	832	1,510	486
Effluent	900	785	365
Total Suspended Solids, mg/l			
Influent	218	249	81
Effluent	688	630	250
Total Vol. Suspended Solids, mg/l			
Influent	147	199	72
Effluent	646	532	241
BOD, mg/l			
Influent	820	1,140	840
Effluent	570	350	250
% Removal	30	69	70
BOD Loading, lbs BOD/day/1000 cu ft	15.1	18.3	38.5
COD, Influent, mg/l	1,520	2,770	1,960
Ratio of BOD to COD, Influent	0.54	0.41	0.43

periods. Thus, it was apparent that the degree of treatment being provided was quite variable and insufficient to produce a satisfactory effluent.

Considerable difficulty was encountered in determining the BOD of the raw and treated wastewater. Because of possible toxic compounds present in the waste, BOD samples were seeded. The seed material was developed in the laboratory from mixtures of river water, primary and final clarifier effluent from the Lincoln municipal wastewater treatment plant and mixed liquor from the treatment plant serving Dorsey Laboratories. In addition to the samples prepared for 5-day BOD determinations, samples were also prepared for "continuous monitoring". Some samples were also set up using the Hach manometric BOD apparatus.

The dissolved oxygen concentration of the "continuously monitored" samples was determined by a Model 5420 BOD bottle probe and Model 54 dissolved oxygen meter manufactured by the Yellow Springs Instrument Company. Data obtained from the "continuously monitored" samples were used to correct for possible time lag and nitrification in the 5-day BOD samples. Even with seeding, lag periods up to two days were encountered in some samples. No chemicals were added to inhibit nitrification and, as a result, corrections were required for some of the samples.

The results of the BOD tests generally indicated that the higher the dilution, i.e., the lower the ratio of wastewater to dilution water, the higher the BOD value. This was quite noticiable for the samples run using the manometric method. For example, the BOD values obtained for the influent composite for March 19 by the manometric method and the standard dilution method were 500 mg/l and 840 mg/l, respectively.

This is indicative of a waste containing inhibiting substances. This is substantiated by the lag period for some of the seeded samples. It also points out the difficulty in obtaining reliable BOD data for industrial wastewaters, particularly where inhibiting substances are involved or the characteristics of the wastewater are quite variable.

LABORATORY STUDY - TREATABILITY OF THE WASTEWATER

The field study on the treatability of the wastewater from Dorsey Laboratories was complicated by the variability in the raw wastewater characteristics and in the hydraulic and organic loadings on the treatment unit. As a result, an extended aeration treatment unit was set up in the laboratory where close controls could be maintained on its operation.

Laboratory Equipment and Determinations - The laboratory treatment system consisted of a Biooxidation Unit manufactured by BioDevelopment Associates of Austin, Texas, two variable speed Sigmamotor pumps, a compressed air supply and a refrigerator which contained the feed solution and the effluent from the unit. A magnetic stirrer provided continuous mixing of the feed solution. Normally only one pump was operated, but during a portion of this study, two feed solutions were fed simultaneously.

The Biooxidation Unit is divided into two chambers by a vertically adjustable baffle. During this study modifications were made on the baffle to improve the flow pattern in the aeration chamber and to prevent the concentrating of solids in the clarification chamber. The aeration chamber has a liquid capacity of approximately six liters and the clarification chamber has a capacity of approximately two liters.

The liquid level in the unit is controlled by an adjustable overflow weir in the clarification chamber. A submerged porous diffuser in the aeration chamber is used to supply air for mixing and oxygen transfer.

The effluent from the unit was monitored for BOD, COD and solids determinations. All tests except for manometric BOD determinations were performed in accordance with the procedures outlined in Standard Methods for the Examination of Water and Wastewater[3]. In addition, samples of mixed liquor were examined with a Leitz orthoplan microscope and photomicrographs were taken with an attached Leitz orthomat camera. Photomicrographs were taken of unstained samples using phase contrast. Mixed liquor for the start-up of the unit was obtained from local activated sludge treatment systems.

Procedures and Observations - The laboratory unit was operated continuously for 114 days. Feed for the unit was obtained from a one-day composite of the raw wastewater from Dorsey Laboratories. The composited wastewater was frozen in plastic containers until just prior to its use. The BOD, COD and total solids values for the composited wastewater sample were 840 mg/l, 1380 mg/l and 697 mg/l, respectively.

The composited wastewater sample was fed without dilution. Feed rates of 1.2, 2.4, 3.0, 3.6 and 4.8 liters per 12 hour feed period were used. A feed period of 12 hours per day was used in order to simulate field loading conditions. Initially the unit was operated at the low feed rate. This was gradually increased up to the 4.8 liter per day feed rate by the 50th day of the run. On the 54th day of operation the sludge volume index (SVI) was 645 and suspended solids were

being carried over the effluent weir. The feed rate was then reduced to 3.0 liters per day without any noticeable change in the SVI.

At this point, the feed solution was changed to primary clarifier effluent from the Lincoln wastewater treatment plant. This waste had a BOD of 250 mg/l and the feed rate was 3.6 liters per day. After seven days of operation using this feed solution, one-half of the activated sludge was wasted. After 22 days of operation the SVI was still 495. At this point, one-third of the activated sludge was wasted - one-half of which was replaced with activated sludge from the Lincoln plant. After 15 additional days of operation, the SVI had dropped to 160.

The feed arrangement was then changed to 4.0 liters per day of primary clarifier effluent and 0.12 liters per day of 10,000 mg/l concentration sucrose solution. This provided an approximate BOD contributory relationship of two-thirds for the sucrose solution and one-third for the primary clarifier effluent. Ammonium chloride was added daily to insure an adequate supply of nitrogen. After seven days of operation using this feed mixture, the system again failed and the run was discontinued. The SVI at the time of failure was 633.

During the run, microscopic examinations were made regularly of the activated sludge floc. These examinations showed that as the initial feed rate of wastewater from Dorsey Laboratories was increased, hair-like filamentous growths developed and emanated from the floc particles. The filamentous growths were identified as Sphaerotilus natans. The rate of growth of S. natans increased sharply at the higher feed rates. Increasing concentrations of S. natans resulted in higher SVI and ultimate failure of the treatment system.

The microscopic examinations also showed that the high concentrations of S. natans persisted for some time after the feed was changed to primary clarifier effluent. The subsequent addition of a sucrose solution feed resulted in a very rapid increase in the concentration of S. natans and in the SVI.

During the run the pH in the aeration chamber ranged from 7.6 to 8.4, the temperature ranged from 20° to 25°C, and the dissolved oxygen concentration ranged from 4.5 to 8.0 mg/l. COD values for the effluent ranged from 30-194 mg/l during periods when suspended solids were not being carried over in the effluent. The COD removal efficiencies ranged from 86 to 98 percent.

Discussion - The results obtained in the laboratory indicated that the composition of the wastewater from Dorsey Laboratories was conducive to the growth of Sphaerotilus natans. The presence of excessive growths of S. natans has undoubtedly been one of the contributory factors to the operational problems encountered in the existing wastewater treatment system at Dorsey Laboratories.

Stokes[4] reported that glucose, galactose, sucrose, maltose, mannitol, succinate, fumarate, butyrate, butanol, glycerol, sodium lactate, sodium pyruvate and ethanol were all found to be good sources of carbon and energy for Sphaerotilus natans. Lackey and Wattie[2] reported that the addition of monosaccharides and disaccharides stimulated the growth of S. natans in normal activated sludge. Many of the products produced at Dorsey Laboratories contain relatively high concentrations of mono-and disaccharides.

Nitrogen requirements of S. natans can reportedly be satisfied by various inorganic salts or organic nitrogen.[4] However, there is some

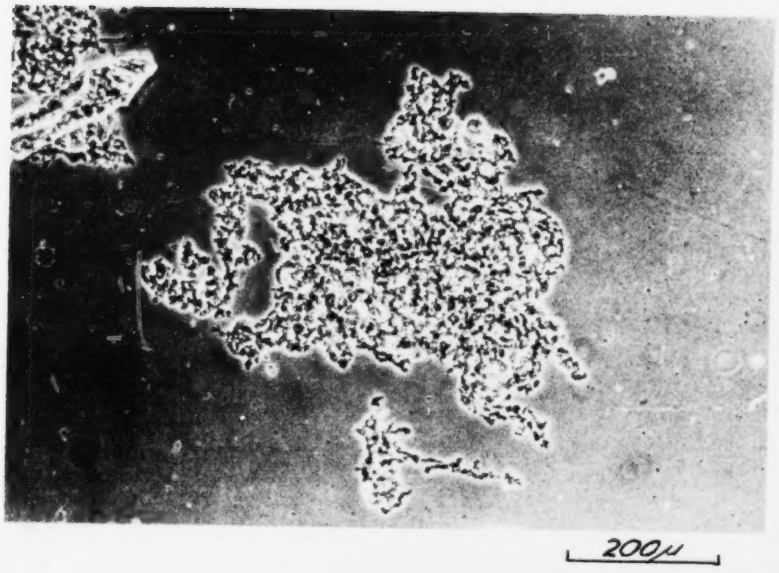
indication that organic nitrogen is preferred.[2,4] Reportedly, for a given concentration of nitrogen, S. natans is capable of utilizing a much higher concentration of glucose.[1] Nitrogen determinations made on the raw wastewater from Dorsey Laboratories indicated a possible nitrogen deficiency during certain periods. Values obtained for ammonia nitrogen ranged from 2.2 to 7.4 mg/l. Organic nitrogen values ranged from 0.2 mg/l to 36.5 mg/l.

One of the distinctive features of S. natans is the containment of the cells in an organic tube or sheath.[4] These sheaths emanate from the activated sludge floc thereby increasing the effective size of the particle and decreasing its specific weight. They may also tend to trap air bubbles. As a result, the sludge has poor settling characteristics and tends to bulk.

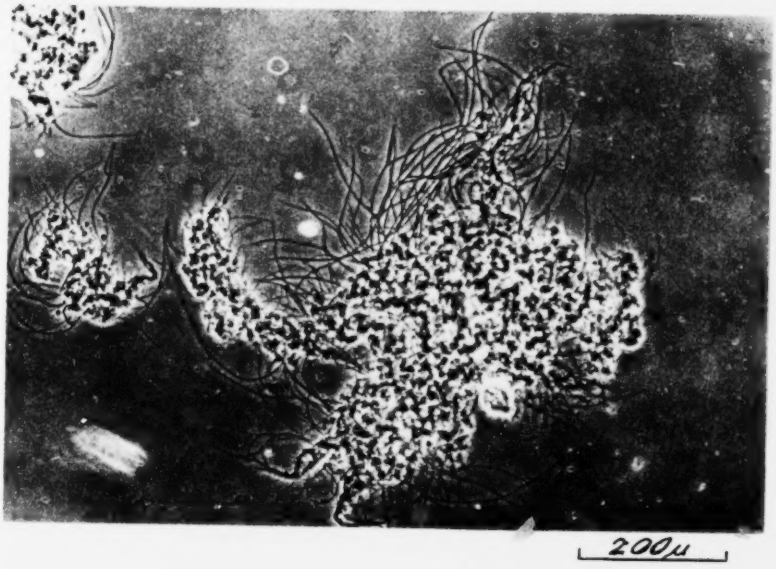
Figure 1 shows two photomicrographs which were taken during this study. The one photomicrograph shows normal activated sludge floc prior to treating wastewater from Dorsey Laboratories. The second photomicrograph shows the filamentous floc obtained from treatment of this wastewater. The probable effect of the filamentous growths on the settling characteristics of the sludge may be visualized from a comparison of the floc in the two photomicrographs. Figure 2 includes a photomicrograph which shows a filament or strand of Sphaerotilus natans in greater detail.

FIELD EVALUATION OF THE TREATMENT SYSTEM

Preparation and Procedure - With this background information, the third phase of the study was initiated. This consisted of a comprehensive evaluation of the existing extended aeration treatment system at



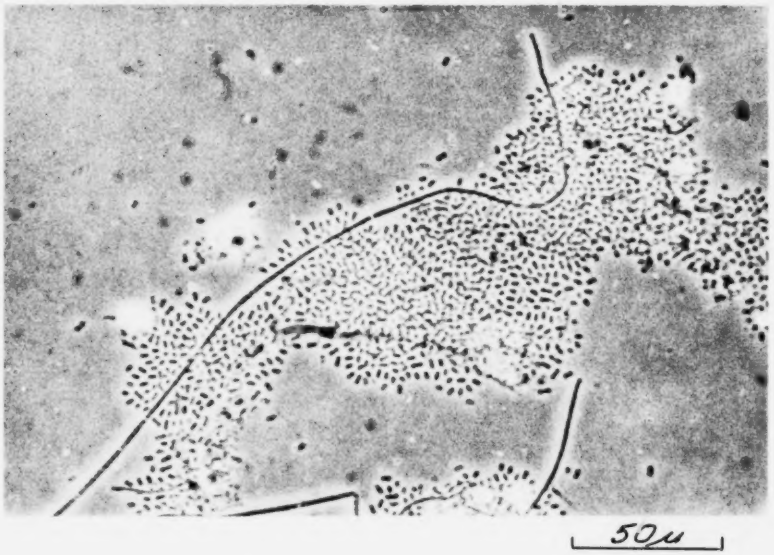
Photomicrograph 1 - Normal Activated Sludge Floc Prior to Treatment of Wastewater from Dorsey Laboratories.



Photomicrograph 2 - Typical Filamentous Floc Resulting From the Treatment of Wastewater from Dorsey Laboratories.

Figure 1 - Photomicrographs of Normal and Filamentous Activated Sludge Floc.

Photomicrograph 3 - Sphaerotilus natans in Filamentous Activated Sludge Floc (Stalked Protozoa are Shown in the Lower Center).



Photomicrograph 4 - Dispersed Bacterial Growth After Treatment of Wastewater from Sulfa Drug Processing.

Figure 2 - Photomicrographs of Sphaerotilus natans and Dispersed Bacterial Growth.

Dorsey Laboratories. Preparatory work included the construction of a flow splitter box ahead of the treatment system. This permitted the diversion of all or controlled portions of the flow around the treatment unit. Also, the aeration tank was drained, cleaned and partially filled with activated sludge from the Lincoln plant prior to the start of the investigation.

This phase of the study ran from May 26, to September 30, 1969. During this period, the system was monitored under different loading conditions and eight one-day composites were made on the system. Data relative to the composites are shown in Table II and Figure 3.

Flow loadings on the field treatment system ranged from 25 percent of the normal flow during the initial portion of the study up to 100 percent of the normal flow during the final 74 days.

Observations and Discussion - The results of the field study confirmed the findings in the laboratory. During the initial 17 days of the study, the discharge of high strength sugar wastes to the system was limited and the system appeared to be functioning normally. However, at that point production of cough syrup commenced and the resulting high strength sugar wastes resulted in an increase in the Sphaerotilus natans concentrations in the activated sludge.

In an attempt to prevent failure of the treatment system, Dorsey Laboratories agreed to keep the high strength sugar wastes out of the wastewater treatment system for the 30 day period corresponding to the 42nd to the 72nd day of the study. This resulted in a noticeable decrease in the concentration of S. natans. However, operational problems were encountered due to insufficient return of solids to the aeration basin. At the end of the thirty day period, high strength sugar wastes

Table II

Composite Data - Final Field Study

	Date of Composite - 1969							
	June 12	June 25	July 15	July 31	Aug 6	Aug 14	Aug 21	Sept 30
Flow, gal/day	4,000	5,200	9,300	18,000	18,900	6,000	25,900	4,800
max. rate gal/day	10,000	10,500	22,000	61,000	95,000	27,000	62,000	21,500
Total Solids, mg/l								
Influent	518	518	471	547	816	2,600	969	2,410
Effluent	445	426	452	478	746	626	564	1,150
Total Volatile Solids, mg/l								
Influent	279	264	178	156	364	2,240	544	1,770
Effluent	190	243	107	147	327	295	169	623
Volatile Suspended Solids, mg/l								
Influent	80	140	48	48	131	203	77	129
Effluent	97	155	42	98	261	105	36	400
COD, mg/l								
Influent	375	378	537	407	681	3,210	662	3,580
Effluent	53	33	90	112	-	206	-	1,344
BOD, mg/l								
Effluent	<3	<9	<9	30	-	50	-	472

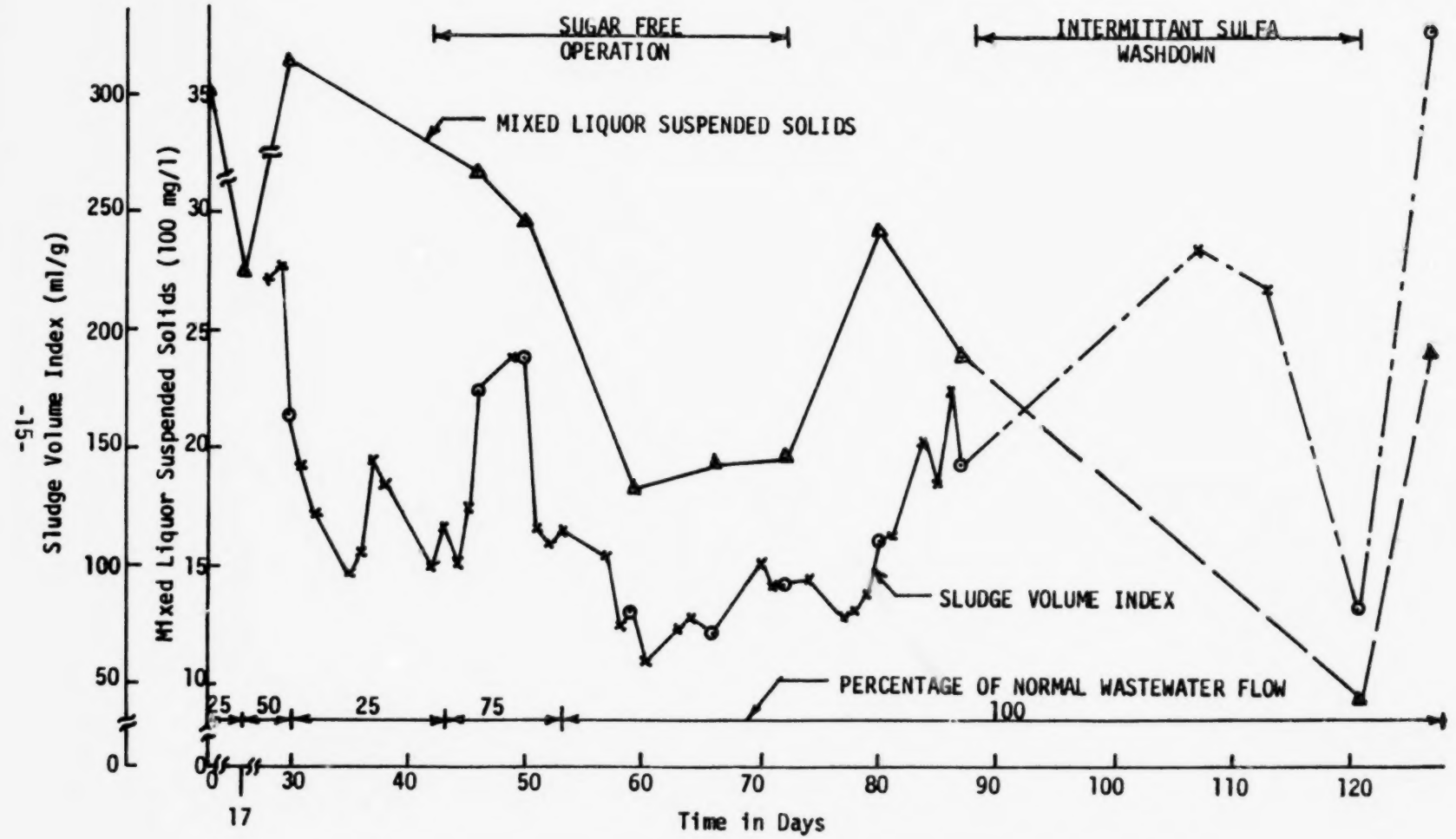


Figure 3 - Mixed Liquor Suspended Solids and Sludge Volume Index vs. Time

were again discharged to the system and a daily application of 12 to 15 lbs of ammonium phosphate to the aeration basin was begun. A rapid increase in the concentration of S. natans was noted after the resumption of discharging high strength sugar waste to the system.

Daily monitoring of the system was not provided during the period from the 77th to the 121st day of operation. A grab sample taken on the 121st day of operation, September 21, showed that the mixed liquor suspended solids concentration in the aeration basin had dropped to $\leq$ 1000 mg/l. Microscopic examinations showed that the floc consisted of dispersed bacterial growths. See Figure 2. Discussions with Dorsey Laboratories revealed that drugs containing sulfa had been produced during this period. During the final six days of the study, no additional sulfa drugs were produced and by September 30, the date of the last composite, the mixed liquor suspended solids concentration had increased to about 2400 mg/l. However, it does point out one of the problems which may be associated with the biological treatment of pharmaceutical manufacturing wastewater, namely the presence of toxic or antibacterial compounds.

Based upon available production figures for cough syrup and an estimated loss of 0.25% of the materials due to washdown, approximately 180 lbs of sucrose is discharged to the wastewater treatment facilities per week during peak production periods. In addition, laboratory tests are made on the sucrose solution and these samples are wasted. This may amount to a shock load to the system of up to 60 lb of BOD. Thus the BOD loading to the system which is associated with the high strength sugar waste probably exceeds 50 percent of the total loading on the system during certain periods of operation.

In addition to the variability in the wastewater characteristics and the organic loading on the system, the hydraulic loading on the system was also quite variable. Flow rates up to 95,000 gallons per day were recorded during the composite periods. This represents a hydraulic loading of approximately 450 percent of design flow. On the other hand, organic and hydraulic loadings on the weekend are essentially zero.

SUMMARY AND CONCLUSIONS

1. The results of the study on the wastewater from Dorsey Laboratories and the existing extended aeration treatment system showed that the existing system was incapable of providing a high degree of treatment for this waste.
2. BOD determinations on the wastewater from Dorsey Laboratories were complicated by the variability of the wastes and wastewater characteristics as well as the presence of inhibiting substances.
3. The relatively high concentrations of mono-and disaccharides in the wastewater from Dorsey Laboratories are conducive to the growth of Spnaerotilus natans.
4. One of the primary reasons for the failure of the system was bulking sludge which was related to an abundant growth of the filamentous bacterium, Sphaerotilus natans.
5. Operation of the wastewater treatment system is further complicated by the extreme variability in the organic and hydraulic loadings on the system, the discharge of toxic or antibacterial compounds in the wastewater and the lack of positive sludge return to the aeration basin.

6. Even though problems associated with the activated sludge treatment of wastewater from Dorsey Laboratories have been identified, there does not appear to be any practical solution at this time. Pretreatment or separate treatment of the high strength sugar wastes is not possible without costly modifications in the sewer piping in the industrial plant.

Results of this study indicate that combined treatment with municipal wastewater is desirable in order to provide dilution of high strength sugar wastes and antibacterial agents as well as a more uniform distribution of hydraulic and organic loadings on the treatment system. In this respect, the City of Lincoln has proposed providing a municipal sanitary sewer to serve this industrial site. However, Dorsey Laboratories may continue to operate their existing wastewater treatment facilities in order to provide pretreatment of the wastes prior to discharge to the municipal system.

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